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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 5 DETERMINATION OF TIN-IODIMETRIC METHOD

(First Revision)

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 5 DETERMINATION OF TIN-IODIMETRIC METHOD (First Revision)

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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 5 DETERMINATION OF TIN-IODIMETRIC METHOD

(First Revision)

0. FOREWORD

- 0.1 This Indian Standard (Part 5) (First Revision) was adopted by the Bureau of Indian Standards on 22 July 1987, after the draft finalized by the Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.
- 0.2 IS: 4027 first published in 1967, covered determination of copper, lead, tin, manganese, phosphorus, nickel, iron silicon, aluminium, zinc and antimony in bronzes. While reviewing this standard, the Sectional Committee decided that it is convenient to revise this standard in series of parts, which, on publication will supersede the relevant method for determination given in 18: 4027-1967*. This part is one of that series and covers the determination of tin by iodimetric method. The other parts are as follows:
 - Part 1 Determination of copper and lead by electrolytic method
 - Part 2 Determination of manganese by photometric method
 - Part 3 Determination of phosphorus by volumetric method
 - Part 4 Determination of nickel by photometric method
 - Part 6 Determination of zinc by complexometric (EDTA) method

Methods for chemical analysis of other constituents in bronzes, namely, aluminium, iron, silicon and antimony are under preparation and will be published in subsequent parts of above series.

- **0.3** In this revision, the figure for determination of tin. incorporated in earlier edition, has been deleted and method has been updated.
- 0.4 The method of analysis prescribed in this standard may primarily serve as referee method and may also be used by the laboratories for

^{*}Methods of chemical analysis of bronzes.

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their day-to-day work. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

0.5 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated; is to be rounded off, it shall be done in accordance with IS: 2-1960*.

1. SCOPE

1.1 This standard (Part 5) prescribes a method for determination of tin ranges as specified in the relevant Indian Standards on bronzes.

Note — This method is not applicable for aluminium bronze and aluminium silicon bronze where tin is 0.20 percent or more.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with IS: 1817-1961†.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, analytical grade reagents and distilled water (see IS: 1070-1977‡) shall be employed in the test.

4. GENERAL

4.1 Use of Filter Paper — In the method prescribed in this standard, relative numbers of Whatman filter papers, which are commonly used, have been specified. However, any other suitable brand of filter papers of corresponding porosity may also be used.

5. DETERMINATION OF TIN BY THE IODIMETRIC METHOD

5.1 Outline of the Method — Tin is separated by precipitation with ammonium hydroxide in presence of iron. After reduction, it is determined iodometrically.

5.2 Reagents

5.2.1 Concentrated Hydrochloric Acid - r.d. = 1.16 (conforming to IS: 265-1976§).

^{*}Rules for rounding off numerical values (revised).

[†]Methods of sampling non-ferrous metals for chemical analysis.

[†]Specification of water for general laboratory use (second revision).

Specification for hydrochloric acid (second revision).

- **5.2.2** Dilute Nitric Acid -1:1(v/v).
- 5.2.3 Ferric Chloride Solution Dissolve 25 g of ferric chloride crystals (FeCl₃6H₂O) in 400 ml of water containing a few drops of concentrated hydrochloric acid and dilute to a litre.
 - 5.2.4 Concentrated Ammonium Hydroxide 20 percent.
 - **5.2.5** Ammonium Chloride Solution 20 g/l (w/v).
- **5.2.6** Antimony Chloride Solution Dissolve 20 gof antimony chloride (SbCl₃) in 500 ml of concentrated hydrochloric acid and dilute to one litre with water.
- 5.2.7 Iron Iron of purity not less than 99.85 percent in the form of wire or strip.
- **5.2.8** Potassium Iodide Solution Dissolve 100 g of potassium iodide in water and dilute to one litre.
- 5.2.9 Starch Solution Make a suspension of 1 g of soluble starch in about 5 ml of water and add it carefully to 100 ml of boiling water. Cool the solution before use. Prepare fresh.
- **5.2.10** Standard Potassium Iodate Solution (0.05 N) Twice recrystallize potassium iodate from water and dry at 180°C to constant weight. Dissolve 1.783 5 g in 200 ml of water containing 1 g of sodium hydroxide and add 10 g of potassium iodide. When dissolution is complete, make up to one litre. Standardize the solution against pure standard tin solution following the procedure described under **5.3.2**.
- 5.2.11 Standard Tin Solution Dissolve 0.5 g of tin in 300 ml of dilute hydrochloric acid (1:1) in 400 ml beaker by warming gently until the metal has dissolved. If the dissolution is difficult, add 0.05 to 0.1 g of potassium chlorate. Cool and make up to one litre.

5.3 Procedure

5.3.1 Transfer 1 000 g of sample into a 250-ml beaker. In case tin content is 0 l percent or less, 10 g of sample should be taken. Add 5 ml of concentrated hydrochloric acid and 20 ml of dilute nitric acid, adding more concentrated hydrochloric acid, if necessary, to keep the tin in solution. When the solution is complete, add 10 ml of ferric chloride solution and boil for 2 minutes. Dilute to 400 ml with water and add concentrated ammonium hydroxide solution until the precipitated copper hydroxide is dissolved and the smell of ammonia persists. Heat to boiling and allow to settle for one hour. Filter through Whatman No. 40 filter paper and wash with hot ammonium chloride solution. Dissolve the precipitate with hot concentrated hydrochloric acid. Reprecipitate with concentrated ammonium hydroxide, boil, filter and

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wash thoroughly with hot ammonium chloride solution. Remove the paper and precipitate from the funnel and place in a 500 ml Erlenmeyer flask. Add 200 ml of water and 75 ml of concentrated hydrochloric acid.

5.3.2 Add two drops of antimony chloride solution and 5 g of iron and swirl the flask to break up the paper and to aid in the solution of the precipitate. Stopper the flask with a rubber bung having two holes one for air condenser and the other for burette. Heat the solution to boiling, and boil with continuous evolution of gas for at least 45 minutes (see Note).

Note - Some undissolved iron should remain in the flask at the end of reduction.

After reduction is complete, cool the contents of the flask to about 10°C maintaining an atmosphere of carbon dioxide by passing carbon dioxide gas into the flask. Add 5 ml of potassium iodide solution and 5 ml of starch solution and titrate with the standard potassium iodate solution to persistent blue colour.

5.3.2.1 Carry out a blank determination following the same procedure and using the same quantities of all the reagents but without the sample.

5.4 Calculation

Tin, percent =
$$\frac{(A-B) \times C}{D \times 10}$$

where

A = volume in ml of the standard potassium iodate solution required for the test solution,

B = volume in ml of standard potassium iodate solution required for the blank,

C = tin equivalent of the standard potassium iodate solution in mg/ml, and

D =mass in g of the sample taken.

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